New Refractory Materials on the Basis of SHS Technology

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Self-propagating high-temperature synthesis (SHS) means the synthesis of materials in a wave of chemical reaction (combustion) that propagates over starting reactive mixture owing to layer-by-layer heat transfer. The systematic investigations of solid-phase metallo-thermal combustion of oxide systems aimed at elaborating new technology of refractory materials production were completed at the Institute of Combustion Problems in Almaty, Kazakhstan.

The solid phase combustion of powder mixtures of refractory chemical elements showing in combustion front propagation all over the sample was found in 1967 by RAS (Russian Academy of Sciences) academician A.G. Merzhanov, V.M. Shkiro, I.P. Borovinskaya [1, 2]. Subsequently the scheme of refractory compound synthesis based on this phenomenon was named "the self-propagating high temperature synthesis (SHS)".

The most important aspect of the SHS attracting constant interest to this phenomenon is the possibility to obtain valuable products of chemical reactions in powders or molded end products directly during the SHS process. One of the most outstanding achievements in the SHS field resulted in synthesis of high temperature superconductors [3, 4].

Now many diverse combined processes on the SHS base are being developed. One of the most promising scientific research trends which has recently got its notable elaboration is a combination of SHS and metallothermy [5]. High temperatures and thermal effects characterize the metallo-thermal oxidation-reduction reactions proceeding in combustion waves. The synthesis temperatures reach up to 1,600-3,000 °C. At such temperatures the refractory oxide compounds and salts being of practical interest as refractory materials are formed. For the first time the possibility of obtaining refractory materials and products by SHS method was shown in [6, 7]. The systematic investigations of solid-phase metallo-thermal combustion of oxide systems aimed at elaborating new technology of refractory materials production were completed at the Combustion Problems Institute and were headed by G.I. Ksandopulo and M.B. Ismailov [8-11]. The above mentioned investigations resulted in development of SHS technology of refractory materials production that now got its wide-scale practical application in lining repairs of high-temperature thermal units. They are: lime stone and cement furnaces, coke and anode mass roasting kilns, roofs of steel melting electric furnaces, open-hearth furnaces and converters, TGP and HGP boiler units, boilers etc.

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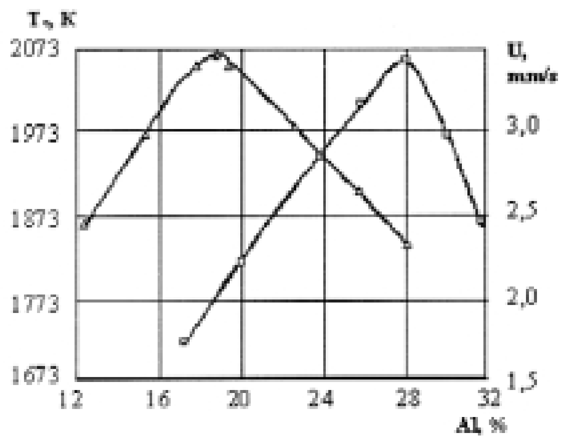

Figure 1 - Combustion temperature and rate in chromite-aluminium system

Solid phase combustion of oxides and natural minerals

Shortly after the discovery of SHS phenomenon, the works on obtaining refractory materials and products were carried out basing on the metallothermy and SHS principles [5-7]. The refractory products were obtained by preparation of exothermal mixture comprising alkali-earth metal chromate, metal-reducer (Al, Mg) and refractory oxide. The mixture was molded in sand or graphite moulds and fired with thermite tablet in air. The use of mineral salts water solutions as a binder makes it possible to prepare masonry mortars, to apply protection coatings on the refractory products.

In view of the change over to cheaper and available raw mate-

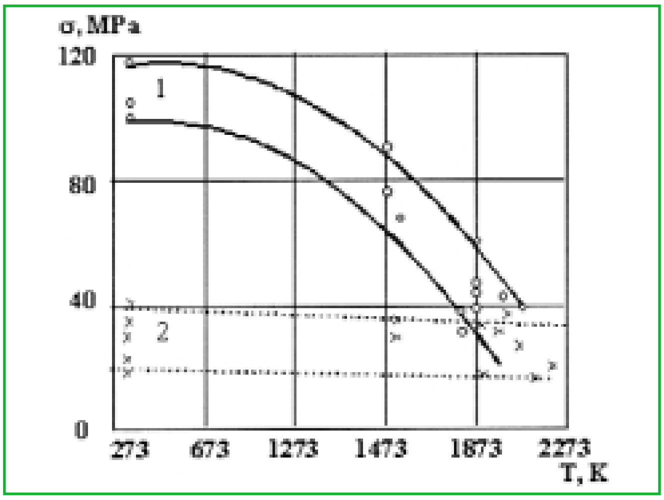

Figure 2 - Dependence of the compression strenght of the SHS product in chromite-aluminium system (1), in chromite-periclase brick (2)

rials in the works of G.I. Ksandopulo, M.B. Ismailov and others [17-20] there were used oxides: Fe_2O_3, Cr_2O_3, B_2O_3, ZrO_2, SiO_2 as oxidants in exothermal mixtures. The systematic investigations of solid phase combustion of pure oxides in SHS conditions were carried out. In such systems the Al, Mg metal powders were used as reducers.

In general form the following chemical reaction may represent the alumothermal combustion of oxides:

$$4/3Al + 2/mMe_nO_m \rightarrow 2n/mMe + 2/3Al_2O_3 \quad (1)$$

The possible proceeding of chemical reaction (1) is determined by changes of isobar-isothermal potential

$$\Delta G = \Delta H - T\Delta S \quad (2)$$

where ΔH is reaction enthalpy and S is entropy.

The mathematical methods of free-gas combustion with slow retardation result in one-to-one dependence of combustion rate *u* on the combustion temperature T_c [21].

$$u^2 \cong (RT_c^2/EQ)\exp(-E/RT_c) \quad (3)$$

where E is the effective activation energy.

The linear dependencies of ln u/T_c on the inverse temperature enable to determine energies of SHS process activation applied in calculations of stable combustion limits of the system.

Diverse combustion conditions: auto-oscillatory (impulsive), spined and stable were found for gasless solid phase combustion [13-14]. Surely, in practical application the stable combustion regime resulting in targeted SHS products is the most interesting.

In paper [15] the combustion in chrome(III) oxide-aluminium system was investigated with different reagent ratios. To extend concentration limits of combustion the source samples in form of compacted cylinders of 2 cm diameter and 4.5-5 cm height were heated up to 1,000 K temperature. The samples dried in the drying oven at 363 K temperature were placed in the muffle furnace at 1,000 K. The sample and furnace temperatures equalized, the combustion was initiated with a tablet of iron-aluminium thermite. It was found that at $[Al]/[Cr_2O_3] \leq 0.8$ and $[Al]/[Cr_2O_3] \geq 14$ reagent molar ratios no system combustion was noted, while at $[Al]/[Cr_2O_3]=0.8 \div 1.2$ and $10 \div 14$ ratios the pulse combustion took place and at 1.2 to 10 ratios the stable state of combustion was observed.

The results of investigations of pure oxide combustion enabled to approach to the study of solid phase combustion of compounds having substantially more complex compositions: natural minerals and raw materials of mining-processing industry [16].

Numerous high-melting compounds are formed in the products of solid-phase combustion of mineral raw materials, i.e. chromite and iron ore concentrates, refractory clays, magnesial compounds, natural alumosilicates. Such formed compounds are interesting as refractory materials.

In paper [17] the regularities of solid phase combustion of chromite ore were investigated and the physical-chemical characteristics of formed products were determined. The used chromite ore has a complex phase composition: Cr_2O_3 54%, MgO 16.5%, Al_2O_3 8%, FeO 7.5%, Fe_2O_3 6%, SiO_2 4.8%, CaO 0.4%. The dispersion degree was up to 0-20 µm. The aluminium powders of PAP-1, ASD-1, PA-4, APV types were used as reducers. The exothermal mixtures of aluminium and chromite were compacted on the press up to form cylinders of 2.5 cm diameter and 4.5-5 cm height. The dependence of combustion temperature and rate on the composition of PA-4 powder is presented in Figure 1.

Unlike pure chrome oxide combustion the temperature dependence has single maximum. The peak of temperature maximum is shifted with respect to the peak of combustion rate. Such behavior characteristics of chromite combustion temperature and rate are due to multistage pattern of the process in the combustion wave [17]. Really, in the chromite, alongside with reduction of chrome oxide, the reduction of iron and silicon oxides proceeds too. And the most exothermal reactions are iron oxide reduction ones*.* The major chemical reactions in the combustion wave are the followings:

$$Cr_2O_3 + Al \rightarrow Al_2O_3 + 2Cr \quad (4)$$

$$Fe_2O_3 + Al \rightarrow Al_2O_3 + 2Fe \quad (5)$$

$$3FeO + 2Al \rightarrow Al_2O_3 + 3Fe \quad (6)$$

$$MgO + Al_2O_3 \rightarrow MgAl_2O_4 \quad (7)$$

$$FeO + Al_2O_3 \rightarrow FeAl_2O_4 \quad (8)$$

$$3SiO_2 + 4Al \rightarrow 2Al_2O_3 + 3Si \quad (9)$$

$$2MgO + SiO_2 \rightarrow MgSiO_4 \quad (10)$$

The effective energy of combustion activation estimated by (3) was equal to 350 kJ/mol. In one or other degree similar reactions proceed in combustion of other natural minerals.

In Figure 2 the dependence of the compression strength of the product of solid phase combustion of aluminium-chromite ore mixture on the temperature is shown.

As shown in Figure 2 the strength characteristics of the composite are substantially higher than the same ones of chromite-periclase brick. Equally with higher refractoriness, i.e. over 2,000 K, the higher compression strength of the products obtained due to the synthesis process is a very important property of refractory materials.

The characteristics and products of magnesite solid-phase combustion of one of raw materials for magnesial refractory material production were investigated [16, 17].
Decomposition of magnesium carbonate initiated at the temperature over 800 K complicates the magnesite combustion:

$$3MgCO_3 + 4\,Al \rightarrow 2\,MgAl_2O_4 + MgO + C \quad (11)$$

$$MgCO_3 \rightarrow MgO + CO_2 \quad (12)$$

Predominant reaction determining combustion temperature is reaction of carbon dioxide reduction [17]. The combustion products contain magnesium oxide, magnesium-aluminium spinel and carbon, the product melting point being over 2,400 K. It is known that carbon containing refractory materials have higher slag resistance in metallurgical productions. So thus, the magnesite SHS products may be of interest as refractory materials for metallurgy.
Refractory materials are also obtained in alumo-thermal combustion of serpentinites. The maximum combustion temperatures reach to 1,873 K. The serpentinite combustion is interesting for the temperature of serpentinite decomposition and SHS initiating being approximately within one range from 1,123 K to 1,223 K. The active silicon oxide formed in serpentinite decomposition process quite intensively reacts with aluminium. The characteristics of temperature changes and serpentinite combustion rate depending on the mixture content of aluminium powder are similar to those observed in silicon oxide combustion process. Mainly the spinels showing higher melting points over 1,873 K are available in the products of serpentinite and aluminium interactions.
In paper [18] the investigation of solid phase combustion of alumosilicates-refractory Arkalik clay and mullite-corundum mortars was carried out. The alumo-silicate compounds serve as a base of the most applied types of refractory materials. The experiments were completed using samples prepared from the mixtures of alumo-silicates and PA-4 aluminium powder granulated in cylinders of 2.5 cm diameter and 5 cm height and in cubes measuring 2x2x2 cm compacted under 5 MPa pressure. The clay and mortar dispersion degree was 10-30 µm.
The linear dependence of $\ln u/T_C$ on the $1/T_C$ characteristic of solid phase combustion enabling to determine effective energies of process activation are shown according to (3).
Two sections of different activation energies are strongly marked on the curve of combustion rate dependence on the temperature. The combustion activation energy of clay-aluminium system within curve section for aluminium content n=0.74±1.11 achieved 170 kJ/mol, while it being equal to 19.8 kJ/mol within section of n=1.11±1.78. Similar results were also obtained for mullitocorundum-aluminium system where activation energy was up to 117 and 12 kJ/mol. These results prove that in first section with lower aluminium content slower reactions proceed comparable with reactions of self-diffusion and reaction sintering of high melting oxides activation energies of which usually exceed 160 kJ/mol. It is within the first curve section that maximum combustion temperatures up to 2,173 K are observed.
Within the second curve section notable for higher aluminium content there are proceeding high-rate reactions with low effective activation energy and at substantially lower temperatures down to 1,400 K. The reactions of aluminium oxidation by air oxygen are additional reactions within this curve section.

The fundamental investigations of solid phase combustion processes, including SHS processes, in oxide systems carried out recently enabled to develop refractory materials in the form of exothermal mixtures useful for repair of heat units [19-28].

New carbon-containing refractories

Within the study of mineral feedstock over-carbonization processes during thermocatalytical pyrolysis, a possibility of carbon-containing refractory materials production that can be applied in metallurgy has been investigated. Obtained materials may be used in the production of refractories, among others also including the SHS technology.
Produced SHS-refractory materials with the addition of carbonized chromite sludge contain metal carbides and elementary carbon and are of better operational characteristics than other similar refractory materials.
The problem of chemically stable refractory is typically solved by the application of either refined oxide systems, e.g. corundum, or carbon-containing refractory materials like resin-magnesite ones and even pure graphite blocks.
One of the most critical matters in carbon-containing refractory production is addition of the carbon to a refractory material.
It has earlier been determined that thermo-catalytic pyrolysis of hydrocarbons on mineral feedstock leads to the formation of carbon fibres of various morphology and structure and with typical properties and physical-mechanical parameters [29, 30].
In this connection application of carbon-and-mineral compositions as refractories was actual. Chromite sludge from Donskoi Mining Plant (GOK) was used as a mineral component of mining industry wastes.
Over-carbonization was conducted at the flowing pyrolysis installation within the temperature range of 723 to 1,173 K and propane-butane mixture consumption $6{,}8{\cdot}10^{-3}$-$7{,}8{\cdot}10^{-3}$ h^{-1}. Sludge blend prepared by extrusion on the granulator was first dried at room temperature, then left for 2 hours in the muffle at 723 °C and finally cooled in the air to room temperature. Thus produced granules were applied in further work. Carburized material was used as one of the components for SHS-refractory like Furnon-3HP mortar produced at the Pilot Production Unit of the Institute. After the synthesis this material was studied for refractoriness using conventional techniques and slag-resistance by the technique developed by the SHS-Refractory Laboratory of the Combustion Problems Institute.
To identify slag-resistance, 2 cm arris cubes have been prepared from carbon-containing SHS-refractories based on chromite sludge wastes. Cubes were put into an oxygen convertor slag that had been first melted in the electrode graphite melting pot. Heating was done in a Tamman-type high-temperature electric resistance oven with the heater made of

Figure 3 - Fibrous carbon on the surface of granules after pyrolysis

a carbon-carbide-silicon material. Temperature during trial was up to 1,300-1,500 °C. The temperature was monitored by Promin optical reference pyrometer. Slag-resistance parameters were registered as either the time of cubes complete dissolution or slag penetration depth within certain period of time and at same temperatures.
In the course of carburization the carbon is accumulated both in the pores of the material and on its surface in the form of fibrous carbon with graphitized structure shown in Figure 3.
Also, the results of electronic microscopy, X-ray phase analysis, micro-diffraction and electron paramagnetic resonance spectroscopy have identified the presence of metal carbides such as Cr_3C_2, Cr_7C_3 etc. in the material. Of interest is the fact that chromium oxide CrO_3 - a toxic compound - is absent in over-carbonized products. There evidently occurred reduction of the following type:

$$3C + 2CrO_3 = 3CO_2 + 2Cr \tag{13}$$

Thus there transition of active chromium oxide CrO_3 into practically inert and biologically inactive metal occurred, consequently application of chromite sludge allows to utilize mining wastes and decrease the content of toxic compounds found in GOKs' dumps.
Obtained over-carbonized sludge is important as one of SHS-refractory blend component. In accordance with the research conducted at the Russian Academy of Science Institute of Structural Macrokinetics (ISMAS) [31], when a combustion wave goes through carbon-containing components and graphite and mineral components, due to favorable conditions, metal carbides constituting mineral raw materials are being formed. Due to very high dispersity of formed carbon it's sufficiently reactive to participate in carbide forming reactions. This results in a composite material framed with mineral compounds such as spinels, carbides and oxides and space between them filled with carbon. Evidently carbon-containing components of refractory materials being inert to slag and metal melts substantially slow down destruction. Besides, decrease in porosity leads to better strength characteristics. Comparison of physical and mechanical characteristics is given in Table 1, which shows that adding of over-carbonized chromite sludge results in increased density and mechanical strength compared with the refractory called "Furnon -3 HP" and insignificant increase in refractoriness up to 1,800 °C.
Of special interest is the use of cheap natural raw material, clay, for the production of refractories with addition of carbon components. Data of the Tchilik clay application is given in Table 2.
Improvement of physical and mechanical indices of carbon-containing refractory materials based on clays and chromite sludge is explained by the formation of fibrous carbon with graphitized structure penetrating into the pores of a refractory material, formation of metal carbides and metals reduction (Fe, Cr) proved by means of electronic microscopy, X-ray structural analysis, micro-diffraction and electron paramagnetic resonance spectroscopy. Slag-resistance data of synthesized carbon-containing refractory material with addition of over-carbonized chromite sludge is given in Figure 4.
Obtained refractory materials have high slag-resistance and chemical stability. Simultaneously the properties typical for chromite-containing refractories showed insignificant growth. This will enable forecast of such comparatively cheap and available refractory material application in high-temperature metallurgical processes of metal production, including precious ones, and also in the production of metal carbides for special purposes (Nb, Ta etc.) and for burial of radioactive wastes.
Application of over-carbonized sludge compared to graphite or coke has a few advantages:
- an even formation of catalytic hydrocarbon is achieved along total surface and due to high gas penetration inside the sample itself each particle is covered by carbon;
- due to the formation of ultra dispersed carbon particles with metal inclusions (300-3,000) they are more chemically stable at SHS.

Monitoring of over-carbonization processes by thermocatalytic pyrolysis will enable production of SHS-refractories with assigned physical and mechanical parameters and operational characteristics sludge.

Table 1 - Physical and mechanical characteristics of refractories with addition of over-carbonized chromite sludge

Refractories	*Ignition temp. K*	*Combustion temp. K*	*Shear σ MPa*	*Porosity %*	*Refractoriness °C*
Additives C + Chromite sludge, %					
1	1,103	1,573	9.6	30.1	1,770
2	1,113	1,673	22.8	16.8	1,770
3	1,203	1,763	28.4	13.5	1,780
4	1,193	1,723	16.4	23.0	1,780
5	1,173	1,653	13.2	27.0	1,800
"Furnon-3HP"	1,100	1,800-1,900	11	25-35	1,770

Table 2 - Physical and mechanical parameters of carbon-containing refractory materials based upon tchilik clay

Addition C + Chromite sludge %	*Ignition temp. °C*	*Combustion temp. °C*	*Compression σ Mpa*	*Porosity %*	*Density g/cm³*
0	750	1,400	6	26	4.5
3	800	1,400	6.4	25	5.8
5	800	1,250	7.2	24	6.4
7	900	1,375	8.0	24	4.2
10	900	1,400	9.6	24	3.7

"Furnon" SHS-refractory materials

The simplest refractory materials are mortars, i.e. refractory masonry mortars. As a rule, mortars are composed of two components: finely milled refractory filler and binder.
The mortars have lower refractory and strength charac-

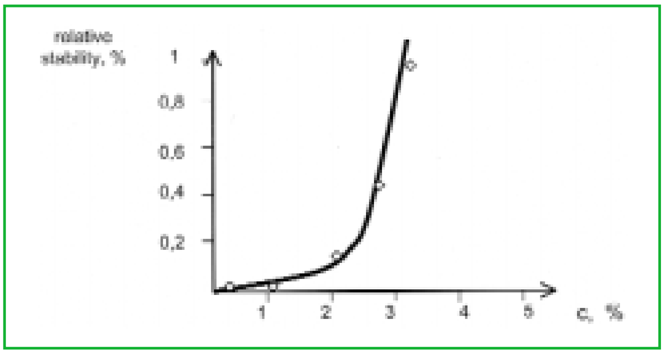

Figure 4 - Dependence of refractory slag-resistance on the concentration of over-carbonized chromite

teristics in comparison with bricks that results in accelerated joint wearing during refractory lining service, being associated with brick splitting-off and falling-out.
The "Furnon" refractory mortars are elaborated and produced by the Combustion Problems Institute using SHS technology.
The high temperatures and huge heat release in SHS process allow to achieve the effective synthesis, sintering and structure formation of refractory compounds, such as corundum, mullite-corundum, spinels, carbides etc. The lining heating up to 1,023-1,373 K temperature on the working surface of thermal unit results in self-ignition of joints burning down to 45-50% lining depth in case of vertical joints. The combustion wave is extinguished from the back of the lining due to heating temperature decrease and heat loss increase in the brick works. The unique property of the "Furnon" mortars is manifested by effect of brick-works "welding" in single whole monolith. This effect is due to the fact that SHS process results not only in the joint material synthesis, but also in chemical interactions between joint material and brick.
So thus, the "Furnon" mortars are gained in operating characteristics through heating process of thermal unit and reaching operation conditions. The above mentioned effects provide high technological adaptability of SHS mortars to be applied as masonry mortars. At the same time the "Furnon" SHS mortars being equal to the best traditional high-alumina and magnesial mortars in refractoriness rates, they prove their superiority over them in high temperature strength of brick binding. At 1,673 K temperature the binding strength of usual mortars achieves 5-7 MPa while the same feature of the "Furnon" mortars being up to 12-15 MPa. In Figure 5 the lining wearing characteristics are shown for cement rotary kiln with usual mortar and "Furnon" mortar applications.
In the past 3-5 years the "Furnon" SHS mortars are widely used in repairs of rotary kilns of many productions. They are: Karaganda cement plant, Kant and Byusmeyin cement plants (Kyrgyzstan, Turkmenistan), lime-stone roasting shift furnaces of Nikolaev alumina plant (Ukraine), OJSC "Vanadium-Tulachermet" (Russia), OJSC "AVISMA-Titanomagnievyi Combinate" (Russia), coke roasting furnaces of Omsk Refinery, anode mass roasting furnaces of aluminium smelting plant in Sibir etc. Positive experience in applications of "Furnon" mortars for linings of transportation and vacuum ladles is accumulated in Bratsk aluminium smelting plant. Here the low wettability of mortars with liquid metal and slag, their chemical inertness to the effects of aggressive media are used rather than refractory properties of SHS mortars. This enabled not only to increase the service life of ladle linings but also to simplify and make easier the process of ladle cleaning, aluminium being discharged.
The effect of bricks "welding" with "Furnon-3ChP" mortar is successfully used in reconstruction of lime stone roasting furnace in Nikolaev alumina plant (NAP, Ukraine). The unique design characteristic of this furnace is that the roasting zone is divided by central core made of periclase-chromite bricks into four sections in each of which the limestone roasting is carried out with gas burners. The design engineering solution of the problem aimed at providing mechanical stability of this most important unit of the furnace became only possible through application of SHS mortar. Two-layered arrangement of eight cyclone burners allows to achieve the uniform and qualitative limestone roasting. The shift furnace of limestone roasting in Nikolaev alumina plant was put in operation in March 1999. The furnace operation is characterized by the following technical features: daily capacity - 145-150 tons, fuel consumption - 150 kg of c.f./ton compared with early 200 kg of c.f./ton, activity of roasted lime - 94% compared with early 85%. The expenditures of the plant were paid back within one year.
The experience in industrial applications of SHS refractory materials proves that 1 ton of SHS mortar used as a masonry mortar makes it possible to save up to 10-12 brick tons only

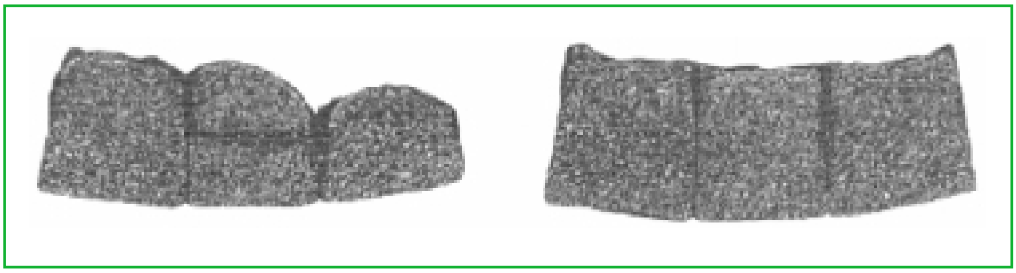

Figure 5 - Wear of thermal unit lining in dependence on the used mortar

Table 3 - Major results in "Furnon" mortar applications

No.	*Brand*	*Applications*	*Results*
1.	Furnon-3ChP	Threshold, cooling and sintering zones of rotary kilns	Increase of threshold and cooling zone service life as much as by 2-3 times and sintering zone - by 1.2-2 times.
2.	Furnon-3	Walls of syphon-pour steel-teeming ladle Chamotte zone of refractory clay roasting rotary	Increase in service life of 3-tons ladles lining as much as by 3 times and of 16-tons ladles - by 1.4 times.
3.	Furnon-7	Hot zone of roasting rotary kilns	Increase of lining service life as much as by 1.8-2 times
4.	Furnon-7Ch	Cold zone of roasting rotary kilns	Increase of lining service life as much as 1.8-2 times

through increase in unit service life. The results of "Furnon" mortar applications are given in Table 3. The economical expediency of SHS refractory applications in specific enterprises is determined by numerous factors, i.e. output of basic production, financial positions of the enterprise, operation conditions of fuel unit, availability of similar material etc. The achieved results and available experience evidence that the SHS refractory applications are very wide and their potentialities are so far from being up.

Conclusion

The SHS refractory mortars are a new generation of refractory materials differing in principles of the traditional ones obtained by "furnace" technology. They are notable for practically complete synthesis of working ceramic body at heating stages of linings through internal energy of components. Further development potentialities of SHS refractory technology in perspective are lying in a number of aspects:
- development of refractory materials having non-traditional phase composition: carbides, nitrides, borides combined with oxides and carbon;
- extended use of wastes of mining and processing industries for purposes of their utilization;
- development of methods and technique of manufacturing molded refractory materials and shaped products of diverse purposes applications.

The absence of lacked somewhat significant refractory production in Kazakhstan and the presence of the most important consumers in the republic promote further intensive investigations and developments in field of refractory technology.

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